

AD-A279 972



REPORT DOCUMENTATION PAGE

1. AGENCY USE ONLY (Leave Blank) 2. REPORT DATE 3. REPORT TYPE AND DATES COVERED
5/27/94 Technical Rept. 7/93-5/94

4. TITLE AND SUBTITLE 5. FUNDING NUMBERS

High Magnetic Field Glow Discharge Ionization Source N0014-87-J-1248

6. AUTHOR(S)

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7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) 8. PERFORMING ORGANIZATION
REPORT NUMBER

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9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) 10. SPONSORING/MONITORING
AGENCY REPORT NUMBER

Office of Naval Research
Chemistry Program
800 N. Quincy St.
Arlington, VA 22217-5660

DTIC QUALITY INSPECTED

11. SUPPLEMENTARY NOTES

To be submitted to the Journal of the American Society for Mass Spectrometry

12a. DISTRIBUTION/AVAILABILITY STATEMENT

12b. DISTRIBUTION CODE

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13. ABSTRACT (Maximum 200 words)

A glow discharge ionization source has been developed which operates inside the high magnetic fields necessary
for a Fourier transform mass spectrometer. Diagrams of the source and a sample spectrum are shown.

14. SUBJECT TERMS

15. NUMBER OF PAGES 3

Elemental Mass Spectrometry, Glow Discharge Ionization,
High Magnetic Fields

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT 18. SECURITY CLASSIFICATION OF THIS PAGE 19. SECURITY CLASSIFICATION OF ABSTRACT 20. LIMITATION OF ABSTRACT

Unclassified

Unclassified

Unclassified

Unlimited



50 94-16657

94 6 3 102



OFFICE OF NAVAL RESEARCH

GRANT # N0014-87-J-1248

R&T Code 4134052

Dr. John Pazik

Technical Report No. 45

High Magnetic Field Glow Discharge Ionization Source

by

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To be submitted

to the

Journal of the American Society for Mass Spectrometry

University of Florida
Department of Chemistry
Gainesville, FL

May 27, 1994

Accession For	
NTIS	CRA&I
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Unannounced	
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High Magnetic Field Glow Discharge Ionization Source

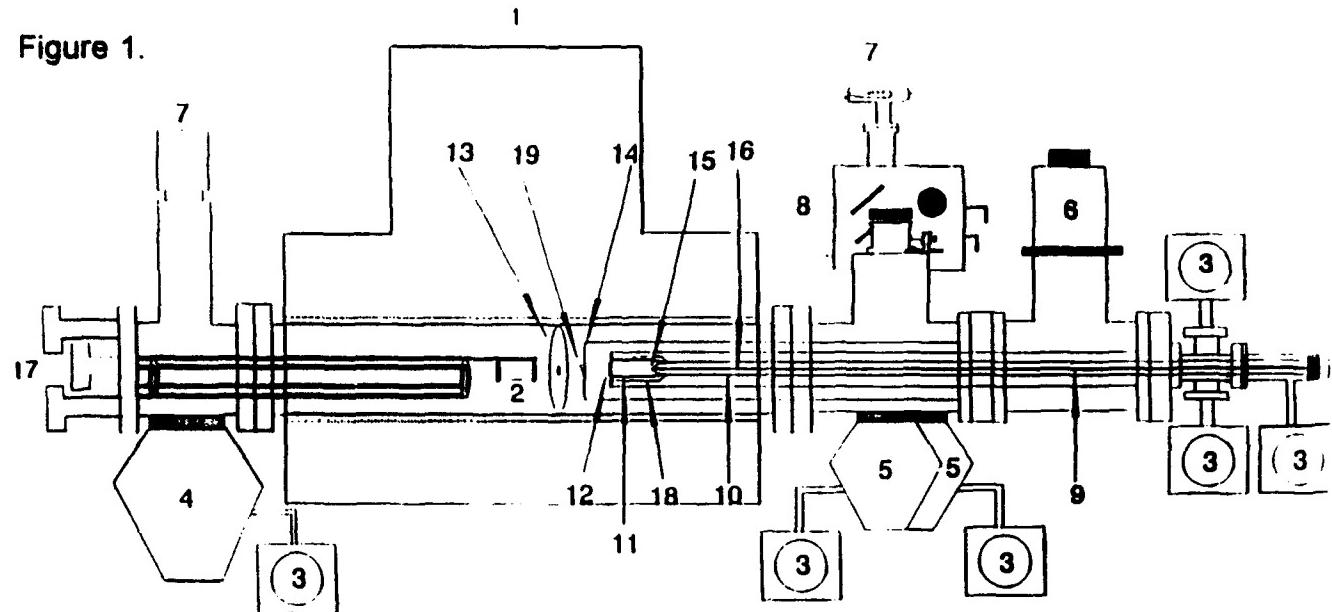
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We have modified our "internal" electrospray source, which operates inside the high magnetic field associated with Fourier transform ion cyclotron resonance (fticr) mass spectrometry, to allow it to serve as a glow discharge (GD) ionization source. Figure 1 shows the overall experimental apparatus for this system, and Figure 2 is an expanded view of the modifications which make glow discharge possible.

Quite reasonable ion signals are obtained with this source, in spite of the fact that the glow discharge ionization is taking place at a magnetic field of about 1.8 tesla. This corresponds to a field at least a factor of 25 higher than has ever been used with glow discharge sources before. One example of an analysis of the $^{60}\text{Ni}^+$ signal in a NIST 661 stainless steel signal is shown in Figure 3. Signal-to-noise calculations for the top trace yield a limit of detection of 38 ppm for the peak at m/z 60. The predominant ion is $^{56}\text{Fe}^+$, arising from the high concentration of iron in the steel. In the middle trace, the main isotope of iron at m/z 56 has been ejected from the icr cell, leading to an improved LOD of 28 ppm for the $^{60}\text{Ni}^+$. In the bottom trace, quadrupolar axialization has been applied to the m/z 60 peak, causing it to be retained in the cell while other ions are (partially) lost due to collisions with a buffer gas. The $^{60}\text{Ni}^+$ LOD is lowered to 6 ppm by this approach.

Preliminary experiments also indicate that the sputter rates for samples in the high magnetic field GD source are a factor of almost 10 lower than those in conventional "external" sources, which do not operate at high magnetic fields. This is promising for the analysis of materials where a large amount of sample cannot be sacrificed, and for analysis of thin films or layered materials.

Figure 1.



The high magnetic field GD/FTICR. (1) superconducting magnet (2T), (2) cell, (3) mechanical pumps, (4) 700 L/s diffusion pump, (5) 300 L/s diffusion pumps, (6) 2900 L/s cryo pump, (7) ion gauge, (8) gas/liquid inlet, (9) high voltage lead, (10) 3/4" stainless steel tubing, (11) Stainless steel mesh, (12) 1 mm orifice, (13) 2nd conductance limit, (14) 1st conductance limit, (15) sample cathode, (16) Ar gas in, (17) laser windows, (18) Delrin insulator and (19) shutter.

Figure 2.

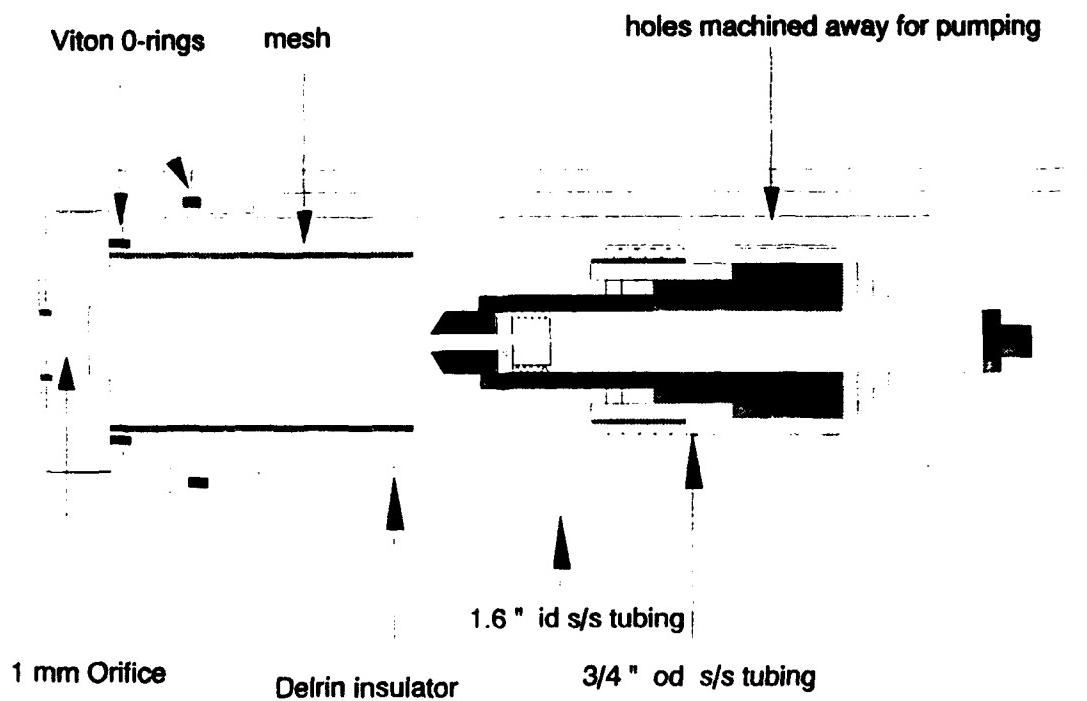


Figure 3.

